



Leaching and drying of herbaceous bales with different density to improve their combustion performance

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ABSTRACT

This study investigates the industrial production of high-quality herbaceous biomass through leaching, using an integrated strategy that combines the use of low-density bales with the avoidance of biomass grinding prior to leaching. Large commercial herbaceous biomass bales ($\approx 2.1 \text{ m}^3$, $\geq 200 \text{ kg}$) were subjected to water leaching in order to improve their combustion performance. Bales with three different densities (100, 140 and 190 kg/m^3) were submerged in 1800 L of water, corresponding to biomass-to-water ratios of 120, 160 and 220 g/L . Leaching duration was investigated, followed by an outdoor drying study over one year. Both bales and leachates were analysed to assess the removal of potassium, phosphorus and other soluble elements, as well as the potential reduction of sintering and corrosion phenomena.

The results show that effective drying of leached bales is only achieved during the warmest summer months ($T_{\text{max}} > 30^\circ \text{C}$). The diffusion of alkaline compounds from straw fibres into water exhibits a conductivity limit of approximately 8 mS/cm , corresponding to about 220 g of straw per litre of water. Bale density strongly influences drying behaviour, and a density of around 140 kg/m^3 is recommended to reach moisture contents below $20 \text{ wt\%}$. Potassium removal of approximately 70% was achieved after 5 h of leaching, which could result in a comparable reduction in fouling and particulate emissions. In addition, corrosion severity was reduced by more than 50% . Although ash melting behaviour improved slightly, deformation and fluid temperatures remained below the thresholds required to fully eliminate sintering risks.

1. Introduction

Given the increasing cost of woody biomass used in biomass-fired thermal plants for heat and electricity production, the search for alternative solid biofuels has become necessary [1]. Herbaceous biomass represents a promising option, as non-woody biomass has significant potential to partially replace fossil fuels and reduce greenhouse gas emissions [2]. Globally, agricultural production generates about 3700 million tonnes of dry crop residues every year. Cereals account for the largest proportion of these residues, followed by maize, rapeseed and sunflower crops. However, only a fraction ($25\text{--}30\%$) of these residues can be sustainably removed because a significant proportion must remain on agricultural land to maintain soil organic carbon, nutrient cycling, and soil structure. Consequently, the sustainable global potential of agricultural residues has been estimated at about 1100 million tonnes per year, providing an important feedstock for bioenergy, advanced biofuels and bio-based products. Within the European Union,

current assessments indicate that the sustainable potential available for bioenergy was estimated at 124 million tonnes per year [3]. At the regional level, Spain produces nearly 8 million tonnes of straw annually, with Castilla y León representing the largest producing region, contributing more than 3 million tonnes each year [4].

However, herbaceous biomass typically contains high concentrations of alkaline compounds, such as KCl , K_2SO_4 or $\text{K}_2\text{Ca}(\text{CO}_3)_2$. These compounds may melt at conventional boiler operating temperatures, leading to slag formation and unplanned plant shutdowns. In addition, volatilised alkaline species can condense on cooler surfaces, such as heat exchangers, causing fouling, clogging and a reduction in thermal and/or electrical efficiency [5,6]. Corrosion is further exacerbated by the aggressive interaction of alkaline compounds with heat exchanger steels. Moreover, particulate emissions increase due to the release of alkaline species as fine particles through the stack, resulting in high flue-gas cleaning costs (e.g. bag filters and electrostatic precipitators) to comply with emission limits [6,7].

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One of the most effective strategies to eliminate or mitigate these issues is the leaching of herbaceous biomass [5–8]. Laboratory-scale leaching studies have demonstrated high efficiency in modifying fuel composition through the removal of water-soluble inorganic compounds [8,9]. This effectiveness is mainly attributed to the intimate contact between water and biomass, particularly when the material is ground to particle sizes generally below 30 mm. At small scale, leaching performance can be further enhanced by extending extraction times, introducing agitation, adding weak acids or increasing the temperature [6,7,9–11]. However, the optimisation of leaching treatments applied to unmilled biomass under pilot-scale or industrial conditions has received very limited attention.

The scientific literature addressing the leaching of whole herbaceous biomass bales (without milling) is very scarce, as most studies have focused on laboratory tests using ground material. To the authors' knowledge, only one previous study has investigated this approach [12], demonstrating that sintering and corrosion risks can be reduced when small bales (17–30 kg) are water-leached. Nevertheless, that study also reported long drying times for bales with densities of approximately 140 kg/m³. Building on this previous work [12], the present study investigates the leaching and outdoor drying of large commercial bales (>200 kg) with different densities (100, 140 and 190 kg/m³) over a one-year period. In contrast to work [12], which employed solid-to-liquid ratios of around 20 g/L—similar to laboratory conditions [7,9,10,13–15]—this work explores substantially higher ratios (≈200 g/L, equivalent to a S/L ratio≈1/5 wt/wt.) in order to reduce water consumption and improve industrial feasibility.

High-density bales (about 190 kg m⁻³) are the most commonly used by biomass power plants. However, bales exposed to rainfall do not dry adequately during summer. Medium-high-density bales (about 140 kg m⁻³) could also require prolonged drying periods, e. g. exceeding 100 days to reduce their moisture content below 20% [12]. To overcome these limitations, this study investigates bales with densities lower (about 100 kg m⁻³) than those currently used commercially, with the aim of determining whether rapid drying to acceptable moisture levels can be achieved within a few days or weeks. Nevertheless, reducing bale density may compromise bale stability and handling characteristics during loading, transportation, and storage. Therefore, an appropriate balance between drying performance and operational feasibility must be established.

Therefore, the objective of this study is to investigate the leaching of large herbaceous biomass bales at high solid-to-liquid ratios as a function of bale density, as well as their subsequent drying, which was the bottleneck in the previous work [12], with the aim of improving combustion performance by reducing alkaline-related emissions and operational issues such as sintering and corrosion. Given the commercial scale of the bales and the high biomass-to-water ratios applied, the results are directly relevant to industrial thermochemical plants and district heating systems.

Furthermore, the potential reuse of the leachate as a nutrient source may offer additional benefits for the fertiliser industry, suggesting a synergistic integration of both sectors. Potassium, the main element leached from biomass, has potential applications in numerous industrial sectors, not only liquid in fertiliser production, but also in soap and detergent manufacturing, biodiesel production, the paper and ceramic industries, as well as pharmaceutical and food applications. The feasibility of these applications depends on the chemical speciation of potassium and its relative proportions in the leachate. In addition, the separation and removal of other leached elements, such as phosphorus, calcium, magnesium, manganese, iron, zinc, chlorine or sulphur require further investigation due to the limited information available with large unmilled biomass bales. Finally, organic extracts from leachate such as sugars and organic acids may have potential for coproduct recovery [16].

2. Materials and methods

2.1. Biomass and commercial bales with different densities

Wheat straw was obtained from a conventional cereal crop placed in Aldeafuente, Soria province (Spain). The straw was left in the field and baled four days after grain harvesting. No rainfall occurred during those four days. At the time of baling, the straw moisture content was approximately 10% (w.b.), and the straw length was around 0.5 m. Wheat straw was harvested at the end of August and processed into rectangular commercial bales. The dimensions of the bales were 2.25 m (length), 0.75 m (height) and 1.25 m (depth), resulting in an individual bale volume of approximately 2.11 m³.

A baler (New Holland, Bigbaler 1290 plus) equipped with a system allowing control of the compaction force applied to the biomass was used to produce bales with different densities. After baling, each bale was weighed and classified according to its bulk density. Three density categories were defined: low density (approximately 100 kg/m³), medium density (approximately 140 kg/m³) and high density (approximately 190 kg/m³). The corresponding bale weights, densities and identification codes are summarised in Table 1.

High-density bales are the most commonly used by biomass power plants and, as discussed in the Introduction, are unlikely to dry adequately after rainfall, even under summer conditions. Consequently, one high-density bale was selected as the reference material in this study. The research investigates bales with lower densities than those currently used commercially to determine whether faster and more effective drying can be achieved. The medium-density bales corresponded to one of the densities evaluated in our previous study [12], although the solid biomass-to-water volume (S/V) ratio in the present work was approximately ten times higher. Low-density bales are expected to dry more rapidly during summer; however, reducing bale density may adversely affect bale stability and handling performance during transportation and storage.

2.2. Leaching treatment in laboratory

To minimise tap water consumption in the subsequent pilot-scale

Table 1
Bale characteristics and experimental leaching conditions.

Bale density and leaching time	Code	Weight (kg)	Density (kg/m ³)	Biomass/water (g/L)	Biomass/water (wt./wt.)
Low density for 1 h	ld 1 h	225	107	125	1/8.0
Low density for 5 h	ld 5 h	201	95	112	1/8.9
Low density for 24 h	ld 24 h	229	109	127	1/7.9
Average		218	104	121	1/8.3
Standard deviation		15	7	8	8
Medium density for 1 h	md 1 h	285	135	158	1/6.3
Medium density for 5 h	md 5 h	281	133	156	1/6.4
Medium density for 24 h	md 24 h	289	137	161	1/6.2
Average		285	135	158	1/6.3
Standard deviation		4	2	2	2
High density for 24 h	hd 24 h	401	190	223	1/4.5

leaching tests (Section 2.3), laboratory-scale leaching experiments were first conducted. Increasing amounts of a representative wheat straw sample were sequentially added at hourly intervals to a fixed volume of water. Portions of approximately 20 g of wheat straw, previously cut to particle sizes below 8 cm, were added to 300 mL of tap water. After each leaching step, the straw was filtered using a quartz filter and the resulting leachate was collected for conductivity measurement. To maintain a constant liquid volume of 300 mL, approximately 50 mL of fresh water was added after each filtration.

A second laboratory test followed the same procedure but with the leaching duration extended from 1 h to 24 h for each straw addition. In this case, approximately 90 mL of water was added after each filtration to maintain the total liquid volume at 300 mL. The conductivity results obtained for both leaching durations (1 h and 24 h) are presented and discussed in Section 3.3.

2.3. Selection of total water volume and leaching conditions in pilot plant

Based on the laboratory leaching tests, a maximum and stable conductivity was achieved at a solid-to-liquid ratio of approximately 225 g of straw per litre of tap water (see Sections 2.2 and 3.3). To maintain this ratio while minimising tap water consumption during pilot-scale leaching, the water volume was determined based on the heaviest bale, corresponding to the high-density bale with a mass of 401 kg (Table 1). This approach resulted in a total water volume of 1800 L.

For this purpose, a commercial swimming pool was used as the leaching vessel (Fig. 1), into which each bale was submerged after filling with 1800 L of tap water. Consistent with the laboratory experiments, pilot-scale leaching durations of 1 and 24 h were selected, while also including an intermediate duration of 5 h. The resulting biomass-to-water ratios for all bales are reported in Table 1.

To ensure a cost-effective leaching procedure, only a partial immersion treatment was applied. Partial immersion was selected to avoid the additional energy required for complete submersion of the bale. Due to buoyancy, the bale floated in the water and was initially submerged to approximately half of its height. As leaching progressed, water gradually diffused upward into the biomass, resulting in a slight increase in the submerged fraction over time. Alternative approaches, such as top irrigation, were not adopted because of the associated pumping cost requirements. To save on irrigation and to enhance water–biomass contact, the bale was turned over halfway through the leaching period. The bale was lifted using a crane and allowed to drain for approximately 30 min to prevent loss of decanted leachate, then inverted with a loading machine and returned to the pool for the remaining leaching duration. Water conductivity in the pool was continuously monitored to control

the leaching process.

Bales were weighed before and after leaching. Biomass samples were collected from the bales using a hook in accordance with the international standard for solid biofuel sampling (ISO 18135:2018). Ten subsamples were extracted from different positions within each bale prior to leaching treatment, and an additional ten subsamples were collected after leaching. The subsamples were homogenised to obtain a representative combined sample for laboratory analysis.

2.4. Bale drying study

After leaching, the wet bales were stored outdoors for one year, from late August 2021 to late August 2022, at the Research Centre for the Development of Renewable Energies (CEDER-CIEMAT), located at 41° 36' N, 2° 29' W in the province of Soria (Castilla y León, Spain). The climate is classified as continental Mediterranean, with cold winters, warm summers, and low rainfall levels. The mean rainfall and maximum, minimum and mean temperatures recorded at CEDER-CIEMAT between August 2021 and August 2022 are shown in Fig. 2. The average relative humidity of the air was 64%, with seasonal variation ranging from 73%, 72%, and 70% in fall, winter, and spring, respectively, to 46% in summer 2022.

Each bale was placed on three standard wooden pallets to enhance air circulation and facilitate drying. The bales were stacked two levels high. An additional unleached bale was placed on a third level and covered to prevent rainwater from affecting moisture measurements. A photograph of the experimental setup is shown in Fig. 3.

Moisture content was periodically determined by collecting ten incremental samples per bale (approximately 3 L each) at different times throughout the year. Sampling was performed using a hook, with five increments taken from one side of the bale and five from the opposite side. The ten incremental samples were combined and homogenised to obtain a representative laboratory sample.

In addition, to monitor the drying behaviour over a one-year period, an unleached bale was placed in one of the four storage piles and designated as the control bale. Consequently, the drying behaviour of a total of eight bales was evaluated: seven leached bales (Table 1) and one unleached control bale. After the first year, all bales were kept under observation for an additional three years to assess their long-term physical stability, until September 2025.

2.5. Physical and chemical tests

Biomass and ash characterisation was performed in accordance with current ISO standards [17–24] for solid biofuels. The analytical techniques and corresponding standards applied are summarised in Table 2.

Biomass ashes were produced at 550 °C following ISO 18122:2015 [18] and subsequently subjected to acid digestion in a microwave



Fig. 1. Swimming pool used for the leaching treatment.

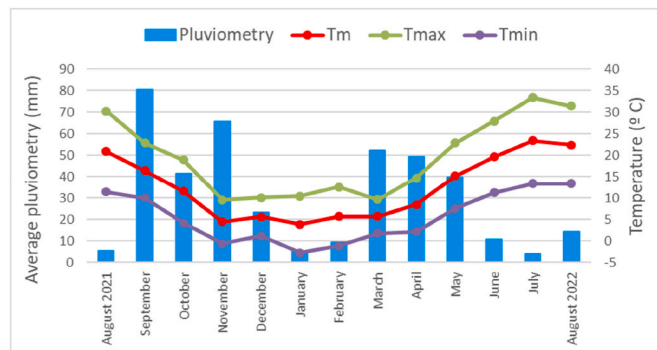


Fig. 2. Average pluviometry and maximum, minimum and mean temperatures recorded at CEDER-CIEMAT between August 2021 and August 2022. Tm: mean temperature; Tmax: maximum temperature; Tmin: minimum temperature.



Fig. 3. Outdoor storage of straw bales for the drying process.

Table 2
Standards and analytical methods.

Property	Analytical method (equipment)	Standard
Proximate analysis	Moisture Drying at 105 °C	ISO 18134-2:2015 [17]
	Ash Ashing at 550 °C	ISO 18122:2022 [18]
Ultimate analysis	C, H, N Elemental analysis (Leco, TruSpec)	ISO 16948:2015 [19]
	S, Cl Ion chromatography (Metrohm, 883 Basic IC Plus) after sample combustion in an automatic calorimeter	ISO 16994:2016 [20]
Calorific value	Automatic calorimetry (Ika, C5000)	ISO 18125:2017 [21]
Major elements in ash	Microwave digestion of the ashes obtained at 550 °C followed by ICP-OES (Thermo Fisher Scientific, Jarrell Ash Iris/AP)	ISO 16967:2015 [22]
Minor elements (As, Cd, Cr, Cu, Ni, Pb and Zn)	Microwave digestion followed by ICP-MS (Thermo Fisher Scientific, iCAP Q)	ISO 16968:2015 [23]
Mercury	Gold amalgamation followed by AAS (Milestone, DMA-80)	ISO 21404:2020 [24]
Ash melting behaviour	Heating an ash pellet to 1450 °C by optical heating microscope (Leitz)	ISO 21404:2020 [24]
Inorganic phases	XRD (PANalytical, X'Pert-PRO)	Not available
Corrosion tests	Biomass ash on steel coupon for 200 h in a tubular furnace at 700 °C and SEM/EDX (Zeiss, Evo LS-10) study	Not available

ICP: Inductively coupled plasma; OES: Optical emission spectroscopy; MS: Mass spectrometry; AAS: Atomic absorption spectroscopy; SEM: Scanning electron microscopy; EDX: Energy dispersive X-Ray spectrometry.

system. Digestion was carried out using HNO₃, H₂O₂ and HF in a first step, followed by H₃BO₃ addition in a second step. The resulting solutions were analysed by ICP-AES (Thermo Jarrell Ash) in accordance with ISO 16967:2015.

X-ray diffraction (XRD) analyses were performed using a PANalytical X'Pert-PRO diffractometer operating at 45 kV and 45 mA. The mineral phases present in the biomass ashes were identified using the Inorganic Crystal Structure Database (ICSD, FIZ Karlsruhe, Germany).

Ash melting behaviour was determined according to ISO 21404:2020

[24] using an optical heating microscope (Hesse Instruments). Four characteristic ash fusion temperatures were reported: shrinkage starting temperature (SST), deformation temperature (DT), hemisphere temperature (HT) and fluid temperature (FT).

In addition, the earth-alkaline to alkaline oxide ratio (I) was used as an indicator to predict ash melting behaviour. This empirical index was previously developed by CIEMAT and successfully applied to different solid biofuels [25,26]. The index I is defined as:

$$I = (\text{CaO} + \text{MgO}) / (\text{K}_2\text{O} + \text{Na}_2\text{O})$$

An increase in the I value is associated with improved ash melting behaviour. In general, ashes with I values higher than 2 are not expected to exhibit sintering risks under typical biomass combustor operating temperatures.

To evaluate the effect of leaching on the corrosion behaviour of biomass ash, laboratory-scale corrosion tests were performed using ashes obtained from the leached bales. Ashes were produced at 550 °C in accordance with ISO 18122:2022 [18] and deposited onto rectangular steel coupons (20 mm × 10 mm, 2 mm thickness), as illustrated in Fig. 4. The selected steel grade was Fe–2.25Cr–1Mo.

Prior to testing, the steel coupons were polished using 220-, 500- and 1200-grit sandpapers and subsequently cleaned with acetone. A fixed amount of ash (50 mg per sample) was placed on each coupon. The samples were arranged in a holder accommodating up to 16 coupons and exposed in a tubular furnace at 700 °C for 200 h under a continuous air flow of 150 mL/min (Fig. 4).

Prior to SEM–EDX (scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy) analysis, the steel coupons were embedded in resin and sectioned using a precision cut-off machine (Struers Secotom-10) equipped with an Al₂O₃ cutting disc and mineral oil as coolant. The cross-sections were subsequently polished using 1200-grit sandpaper and cleaned with hexane in an ultrasonic bath.

Steel thickness and the elemental composition of the metal–corrosion interface were analysed by SEM–EDX using a Zeiss Evo LS-10 microscope.

3. Results and discussion

3.1. Biomass composition

Table 3 summarises the physical and chemical composition of the baled biomass. The reported values correspond to the average of the seven bales analysed. These average values are within the typical range reported for cereal straw. The average ash content was 8.1 wt% d.b., together with relatively high chlorine, sulphur and nitrogen contents, which are characteristic of herbaceous biomass. The average net calorific value was 16.3 MJ/kg d.b., also consistent with values reported for similar fuels.

Silicon was the most abundant ash-forming element (16.1 wt% d.b.), followed by potassium (15.7 wt% d.b.) and calcium (7.0 wt% d.b.), and phosphorus and magnesium present at levels about 1 wt%. The remaining elements were present at concentrations well below 1.0 wt% d.b. Trace element contents were low and were not expected to pose environmental concerns, either in terms of particulate or gaseous emissions through the stack or with respect to ash utilisation, e.g. as

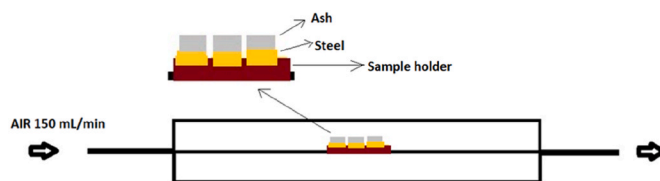


Fig. 4. Schematics of the corrosion test using a tubular furnace (with permission of FUEL journal [12]).

Table 3

Physicochemical properties of baled wheat straw (7 samples). Major ash elements determined after ashing at 550 °C.

Parameter	Unit	Mean	SD	%RSD
Moisture	(% wb)	10.4	1.7	17
Ash	(% db)	8.1	1.3	15
C	(% db)	44.4	0.6	1.4
H	(% db)	5.6	0.1	2.3
N	(% db)	0.76	0.21	27
S	(% db)	0.12	0.02	19
Cl	(% db)	0.49	0.08	17
GCVv	(MJ/kg db)	17.5	0.3	1.6
GCVv	(MJ/kg wb)	15.7	0.4	2.5
NCVp	(MJ/kg db)	16.3	0.3	1.6
NCVp	(MJ/kg wb)	14.4	0.4	2.8
Major elements on ash 550 °C				
Ca	(% db)	7.0	2.6	37
Fe	(% db)	0.077	0.011	14
K	(% db)	15.7	2.3	15
Mg	(% db)	1.22	0.20	16
Mn	(% db)	0.033	0.025	74
Na	(% db)	0.31	0.13	41
P	(% db)	1.23	0.17	14
Si	(% db)	16.1	2.3	14
Ti	(% db)	0.013	0.006	45
Zn	(% db)	0.017	0.006	35
Ash melting behaviour				
SST	(°C)	679	19	3
DT	(°C)	836	25	3
HT	(°C)	1090	30	3
FT	(°C)	1123	24	2
Minor elements				
As	(mg/kg db)	0.14	0.02	13
Cd	(mg/kg db)	0.03	0.02	62
Cr	(mg/kg db)	<1.0	NAP	NAP
Cu	(mg/kg db)	6.1	1.4	23
Hg	(mg/kg db)	0.005	0.001	18
Ni	(mg/kg db)	<1.0	NAP	NAP
Pb	(mg/kg db)	0.34	0.04	11

SD: standard deviation; %RSD: relative SD; db: dry basis; wb: wet basis; GCVv: gross calorific value at constant volume; NCVp: net calorific value at constant pressure; SST: shrinkage starting temperature; DT: deformation temperature; HT: hemisphere temperature; FT: fluid temperature. NAP: not applicable.

fertiliser under European Regulation (EU 2019/1009) or landfill disposal.

For most parameters, variability among bales was low, as indicated by the relative standard deviation (%RSD). Higher variability was observed for nitrogen and calcium, with %RSD values of 27% and 37%, respectively. This variability can be attributed to the heterogeneous incorporation of sand during the baling process, driven by uneven terrain conditions. The sand, being of calcareous origin, contributes significantly to the observed variability in calcium content. Consequently, this variability may lead to deviations in the removal efficiency of calcium and other elements, particularly when comparing bales of similar density, as can be seen in Section 3.4.

3.2. Laboratory leaching tests

Figs. 5 and 6 show the water conductivity results obtained from laboratory leaching tests conducted with leaching durations of 1 h and 24 h, respectively. In both cases, conductivity increased progressively with the amount of straw added until a plateau was reached. Maximum conductivity values of approximately 7 mS/cm were observed for the 1 h leaching tests (Fig. 5), while values close to 8 mS/cm were reached for the 24 h leaching tests (Fig. 6). These results indicate the existence of an upper limit for the diffusion of inorganic compounds from straw stem fibres into the water. Beyond this limit, the addition of further straw did not lead to any increase in conductivity, suggesting that the leaching process is constrained by diffusion-controlled mechanisms.

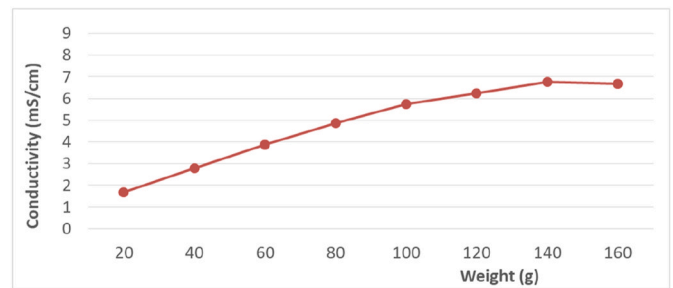


Fig. 5. Electrical conductivity response to straw addition during 1 h leaching.

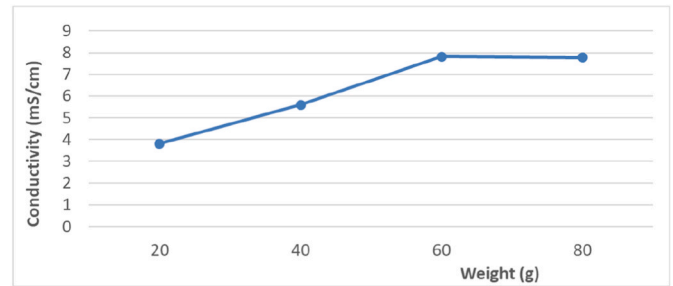


Fig. 6. Electrical conductivity response to straw addition during 24 h leaching.

This conductivity limit was reached with approximately half the amount of straw when the leaching duration was extended from 1 h to 24 h (about 60 g compared to 140 g, Fig. 6 and Fig. 5, respectively). Based on the highest conductivity values obtained in both tests, a maximum solid-to-liquid ratio was estimated. Considering 140 g of straw leached in a total water volume of 622 mL, the corresponding ratio was calculated to be approximately 225 g of straw per litre of tap water.

3.3. Pilot plant scale leaching tests

As indicated by the laboratory leaching tests discussed in Section 3.2, the maximum conductivity achieved (7–8 mS/cm) may limit the fraction of soluble compounds extracted from the biomass into the water. The conductivity results obtained for the bales leached at pilot scale are shown in Fig. 7. The highest conductivity values, close to 8 mS/cm, were observed for the high-density bale after 24 h of leaching. These values are consistent with the maximum conductivity measured under laboratory conditions for this wheat straw and are therefore interpreted as representing the upper limit for the diffusion of soluble inorganic compounds into the leaching water.

Leaching duration had a clear effect on extraction efficiency, as indicated by the higher conductivity values obtained after 24 h

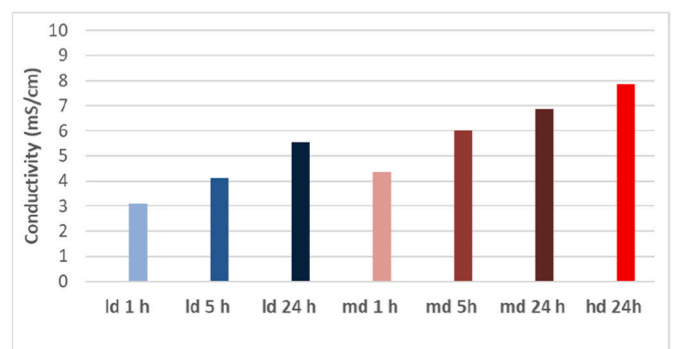


Fig. 7. Conductivity results of the bales tested in the pilot plant. Code bales are shown in Table 1.

compared with those measured after 5 h and 1 h of leaching (Fig. 7). This behaviour is expected to be reflected in the removal of individual elements and inorganic compounds. As ultimately observed (Fig. 8), the general trend in the conductivity data agrees with the element removal results, which show the extraction percentages of the major elements in wheat straw bale ash.

Potassium removals reached approximately 80% for both medium-density and high-density bales after 24 h of leaching (Fig. 8). Other elements associated with highly soluble compounds, such as chlorine mainly present as KCl, showed near-complete removal, with recoveries approaching 100%. In contrast, sulphur exhibited higher apparent removals in low-density bales than in high-density bales. This behaviour may be attributed to the lower solubility of K_2SO_4 compared with KCl, together with enhanced diffusion under lower compaction conditions, which could facilitate the transport of sulphate species in low-density bales.

A reduction of, e.g., 70% in potassium content is expected to result in a substantial decrease in fouling and particulate emissions from the stack, potentially of a similar magnitude. This assumption is supported by the fact that particulate matter emitted during biomass combustion largely consists of alkaline compounds, which volatilise at combustion temperatures and subsequently condense as fine particles. As an example, Fig. 9 illustrates the inorganic compounds identified by XRD in wheat straw bales before and after leaching for the high-density bale leached for 24 h, where the dominant alkaline phases were KCl (sylvite) and K_2SO_4 (arcanite). These estimates of fouling and emission reductions are supported by numerous authors, as highlighted in the review by Gudka [6], and have also been experimentally confirmed, e.g., Khalsa et al. [2]. In addition, Khalsa et al. have also reported that leaching is a suitable approach to producing a competitive solid fuel from grass and foliage.

Regarding the other alkaline element, the sodium content was approximately 50 times lower than that of potassium (Table 3); therefore, its contribution to particulate emissions through the stack can be considered negligible.

Among the three major ash-forming elements (K, Si and Ca), silicon, mainly present as silica (quartz), was not solubilised during leaching. Other elements associated with non-volatile and poorly soluble compounds, such as aluminium and iron, exhibited zero recoveries (aluminium results are not shown in Fig. 8). Up to approximately 15% of the titanium was removed in the leachate. One possible explanation is the formation of a slightly soluble titanium compound in the wheat straw ash, e.g. potassium titanate. However, this hypothesis cannot be confirmed, as no titanium compounds were detected by X-ray diffraction, likely due to their low concentration.

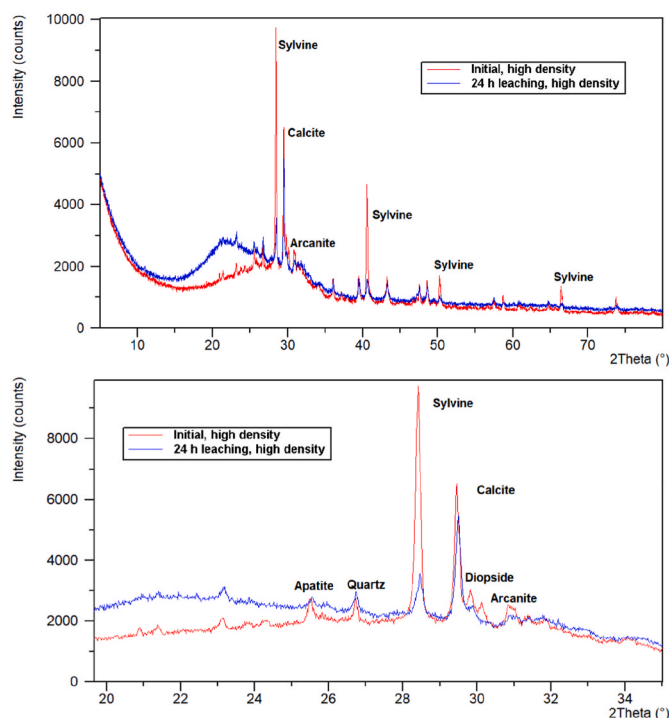


Fig. 9. Inorganic compounds identified by XRD in the high density bale (Upper part: initial and 24 h leaching; Lower part: 30° range with detailed view).

Calcium, another major constituent of wheat straw ash, exhibited unexpectedly high solubilisation levels, reaching up to 50% for the medium-density bale and approximately 35% for the high-density bale after 24 h of leaching. XRD analysis identified calcite as the main calcium-bearing phase, which is generally considered poorly soluble. However, partial dissolution of calcite ($CaCO_3$) was evidenced by the reduction of its diffraction peaks in the XRD patterns (Fig. 9). Phosphorus also showed recoveries of up to 50%, despite being mainly associated with apatite ($Ca_5(PO_4)_3$), another sparingly soluble phase. The lower intensity of apatite peaks in the XRD patterns of the 24 h leached high-density bale suggests partial solubilisation. A similar behaviour was observed for magnesium, which is partly associated with diopside ($CaMgSi_2O_6$), a slightly soluble phase that may account for the observed magnesium removal.

Some results shown in Fig. 8 appear anomalous, for example, for K and Cl in the ld 5 h and ld 24 h treatments, where higher K and Cl

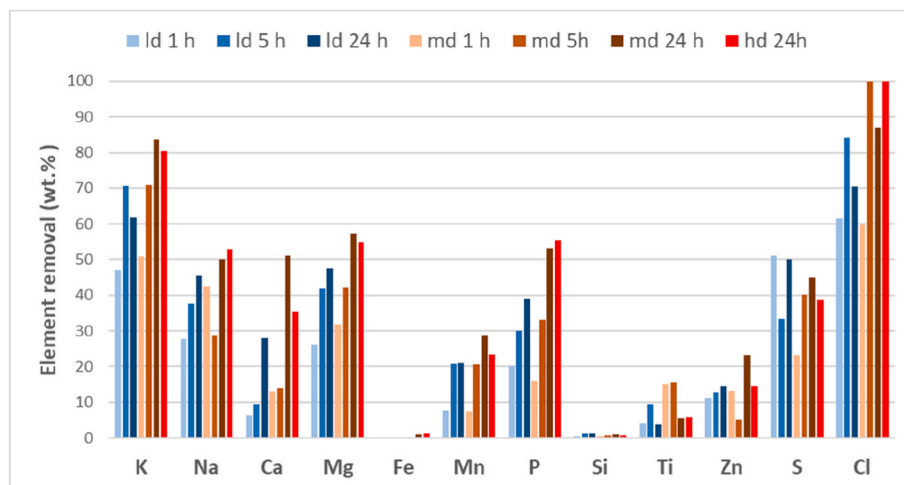


Fig. 8. Removal of chemical elements from straw bales tested at pilot scale as a function of bale density and leaching duration (see Table 1 for codes).

contents were measured after 5 h of leaching than after 24 h, contrary to the expected trend. This behaviour may be attributed to the substantial variability in Ca and other elements among the initial bales, likely resulting from the heterogeneous incorporation of soil-derived sand during straw collection, as discussed in Section 3.1. In fact, the Ca concentration was higher in the 24 h treatment (ld 24 h) than in the 5 h treatment (ld 5 h), which may have caused a relative dilution of other major ash-forming elements, such as K and Cl, in the ld 24 h treatment.

Trace elements in biomass ash, such as manganese and zinc, exhibited removals of up to approximately 20% (Fig. 8). In contrast, copper and lead generally showed removals below 10% (results not shown), while other trace elements, including nickel and chromium, were not quantified due to concentrations below the analytical quantification limits (Table 3). From a nutrient perspective, the leachate may contain essential trace elements for plant growth, potentially including boron, which was not analysed in this study. Consequently, the leachate could be considered for use as a liquid biofertiliser from a mineral nutrition standpoint.

3.4. Effect of leaching on ash melting behaviour

The characteristic ash melting temperatures of all leached bales showed an overall improvement compared with those of the untreated biomass (Table 4). However, in most cases, the increase in these temperatures does not appear sufficient to fully mitigate sintering and slagging risks. Deformation temperatures (DT) generally remained below 1000 °C, and fluid temperatures (FT) were relatively low, around 1200 °C, except for the high-density bale, indicating that ash-related operational issues may still occur under typical combustion conditions.

Deformation temperature (DT) may have limited reliability as an indicator, since severe sintering is generally assumed to already occur at this temperature [25]. To further assess sintering propensity, a predictive empirical index I (earth-alkaline to alkaline oxide ratio) was therefore calculated (Table 4). As previously demonstrated [26], biomasses with I values higher than 2 are generally not expected to exhibit sintering risks at typical combustion operating temperatures (≈ 850 °C). In the present study, none of the leached bales reached I values above this threshold. Consequently, further improvement in ash behaviour would require enhanced element removal, for example, by reducing the biomass-to-water ratio, which would involve increased water consumption.

Other authors, such as Albelha et al. [27], have also reported limited reductions in slagging when leaching/torrefaction is applied at a ratio of approximately 200 g of milled biomass per litre of water. In contrast, their combustion results show several improvements: potassium removal of 60–80%, effective corrosion mitigation, a strong reduction in fine particulate matter, and reduced fouling. These results from Albelha et al. seem quite similar to these ones obtained in this work.

Table 4

Ash melting behaviour of wheat straw bales as a function of bale density and leaching duration (see Table 1 for codes).

Bale code	SST (°C)	DT (°C)	HT (°C)	FT (°C)	(CaO + MgO)/(K ₂ O + Na ₂ O) (Wt.%/wt.%)
Without leaching	679	836	1090	1123	0.60
ld 1 h	710	880	1160	1210	0.79
ld 5 h	700	900	nd	1250	0.70
ld 24 h	700	860	1070	1190	0.80
md 1 h	690	850	nd	1160	1.05
md 5 h	690	840	1070	1180	0.92
md 24 h	700	910	nd	1260	0.97
hd 24 h	750	1090	nd	1440	1.28

SST: shrinkage starting temperature; DT: deformation temperature; HT: hemisphere temperature; FT: fluid temperature. nd: not determined.

3.5. Effect of leaching on ash corrosion behaviour

Changes in metal thickness and oxide layer growth under the conditions described in Section 2.5, as assessed by SEM analysis, were used as indicators of the corrosive behaviour of wheat straw ash. Accordingly, Fig. 10 illustrates the extent of metal corrosion and the corresponding iron elemental mapping for ashes obtained from baled biomass before and after 24 h of leaching, for low-, medium- and high-density bales.

An increase in iron oxide layer thickness was associated with enhanced corrosion of the steel. The oxide layers were primarily composed of iron and oxygen, as confirmed by elemental analysis. Iron elemental mapping for each bale density is shown in Fig. 10. For untreated biomass ash, the oxide layer exceeded 200 µm in thickness, reaching values close to 300 µm. In contrast, leaching substantially reduced oxide layer thickness to values well below 200 µm, typically around 100 µm. Overall, corrosion was reduced by more than 50% as a result of the removal of alkaline compounds during the leaching of wheat straw.

Namkung et al. [28] determined in his corrosion experiments, similar to the one performed in this work, that the reduction in corrosion is due to the absence of elements such as K, S, Ca, P, and Mg, which were not detected on the metal surface. In this work, not only are K, S, and Cl reduced, but also Ca, P, and Mg. The effect of some elements, such as K and S, on corrosion is well-studied, but others, such as Ca and Mg, have more difficulty defining their corrosion mechanisms or are less well-studied [29].

3.6. Bale drying and physical stability

As discussed in the introduction, drying represents the main bottleneck in the implementation of leaching treatments for high-density herbaceous biomass bales. The results of the outdoor drying study are presented in Fig. 11. Low moisture contents (<15 wt% w. b.) were only achieved during the twelfth month of storage (August) for most bales, corresponding to the summer period, when daytime temperatures at the experimental site typically exceeded 30 °C, with mean monthly temperatures around 23 °C (see Fig. 2).

Acceptable drying levels were not reached during autumn (months 1 and 2, corresponding to September and October) or winter (month 5, corresponding to January) for any of the bales tested. Even the unleached control bale exhibited an increase in moisture content from an initial value of approximately 10 wt% to more than 20 wt% by the fifth month of storage (January). This behaviour is attributed to moisture uptake from ambient air during the colder months, characterised by average monthly temperatures of around 5 °C (see Fig. 2) and frequent nighttime frosts.

It is important to note that the high-density bale (hd 24 h) did not reach low moisture contents even after one year of outdoor storage, exhibiting a moisture content of approximately 40 wt% during the twelfth month (August). This result clearly demonstrates that a reduction in bale density is required to achieve moisture contents below, for example, 20 wt% (w.b.), which will ensure the self-sustained combustion of the biomass in a thermochemical facility. In addition, leaching duration significantly influences the initial moisture content of the bales: longer leaching times lead to higher initial moisture levels and, consequently, hinder subsequent drying (Fig. 11).

These findings indicate that not only should bale density be limited to approximately 140 kg/m³, but leaching duration should also be restricted to relatively short periods, preferably between 1 and 5 h, in order to achieve substantial moisture reductions during the summer season.

Nevertheless, drying behaviour must be evaluated together with element leaching efficiency and the reduction of sintering and corrosion in order to draw overall conclusions. These aspects are addressed in the following sections.

With respect to mechanical stability, bale stacks consisting of three

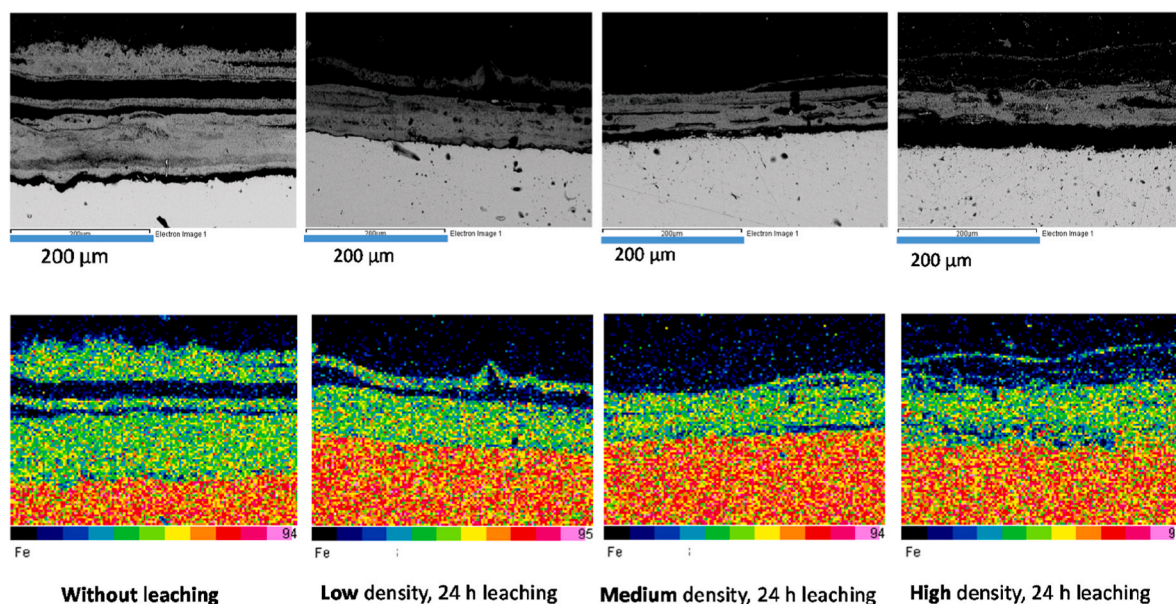


Fig. 10. Metal corrosion (SEM analysis) depending on bale density before and after 24 h leaching.

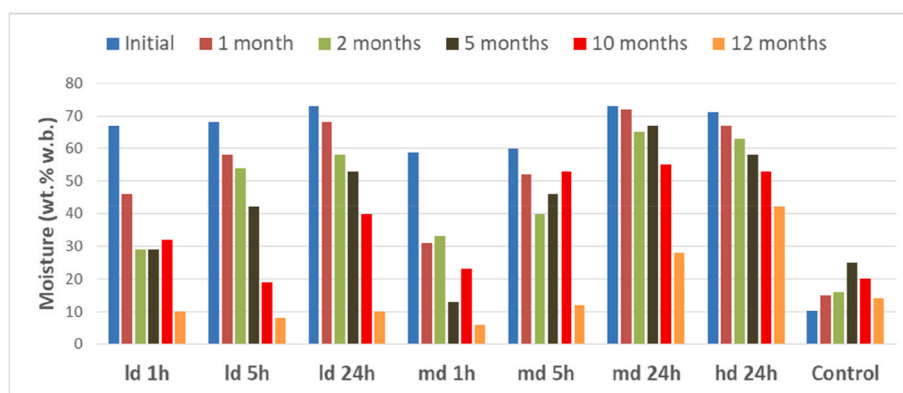


Fig. 11. Effect of the storage time (months) on the moisture content of baled biomass (see Table 1 for codes).

vertically stacked bales (Fig. 3) remained stable for more than one year after initial stacking in August 2021. During the second year of storage (2022–2023), low-density bales progressively lost vertical alignment and collapsed during the third year (2023–2024). In contrast, medium-density and high-density bales remained structurally stable until 2025.

4. Conclusions

Under the conditions tested in this study, effective natural drying of leached herbaceous biomass bales is only achieved during the warmest summer months, when maximum daily temperatures exceed 30 °C. During autumn, winter and spring, drying is negligible.

The ratio between leached biomass and leaching water exhibits an upper limit governed by the diffusion-controlled release of soluble inorganic compounds from straw fibres into water. For the wheat straw investigated, this limit corresponded to a conductivity of approximately 7–8 mS/cm, equivalent to a maximum solid-to-liquid ratio of about 225 g of straw per litre of water (equals to 1/4.4 wt/wt.).

Bale density strongly influences post-leaching drying behaviour. High-density bales ($\approx 190 \text{ kg/m}^3$) could not be adequately dried under outdoor summer conditions, whereas reducing bale density to approximately 140 kg/m^3 enables final moisture contents below 20 wt% to be achieved.

Potassium removal reaches approximately 70% after 5 h of leaching

and increases to about 80% after 24 h. Although longer leaching durations slightly improved extraction efficiency, shorter leaching times substantially facilitate subsequent drying. Consequently, a leaching duration of around 5 h represents a suitable compromise between alkaline removal and drying feasibility. Significant removals of other elements, such as phosphorus, calcium and magnesium, are also observed.

Corrosion test demonstrated a reduction of more than 50% in oxide layer thickness compared with untreated biomass ash, which could indicate a substantial improvement in the corrosive behaviour of wheat straw ash and suggesting an extension of boiler component lifetime. However, despite an increase of approximately 100 °C in fluid temperatures determined according to ISO 21404:2020, sintering and slagging risks still persist.

As a general conclusion, the results of this study indicate that leaching whole herbaceous biomass bales, i.e., without prior milling, at bale densities ranging from 100 to 140 kg m^{-3} , can produce a solid biofuel of sufficient quality for use in facilities such as farm-scale boilers, district heating systems, and biomass-fired power plants.

5. Future perspectives

Further research is required to assess alternative drying strategies, such as forced-air or indoor drying, with or without air heating, in order

to reduce drying times and improve overall process feasibility. In parallel, the production of lower-density bales could mitigate drying limitations, although this would reduce energy density and increase transportation costs. A comprehensive techno-economic analysis considering combustion improvements in sintering and corrosion, particle emission reductions, drying options, water consumption and nutrient recovery (inorganic and organic compounds) from leachates is therefore essential to determine the industrial applicability of leaching treatments for baled herbaceous biomass.

CRedit authorship contribution statement

Miguel J. Fernández-Llorente: Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Resources, Supervision, Validation, Visualization, Writing – original draft, Writing – review & editing. **Ruth Barro:** Formal analysis, Investigation, Methodology, Resources, Validation, Writing – review & editing. **Raquel Bados:** Investigation, Methodology, Resources, Writing – review & editing.

Declaration of interest statement

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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